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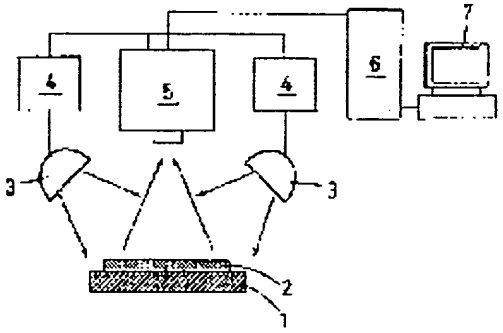
SUGANUMA ETSURO

(54) NON-DESTRUCTIVE EXAMINATION METHOD FOR CARBON FIBER REINFORCED CARBON COMPOSITE MATERIAL

(57)Abstract:

PROBLEM TO BE SOLVED: To detect efficiently and accurately defects in the carbon fiber reinforced carbon (C/C) composite material, by radiating heat ray on the surface of C/C complex, by entering thermal radiation energy from the surface as image using an infrared thermo-graphic device, and by measuring time-dependent variation in the surface temperature distribution.



SOLUTION: C/C composite material 2 to be inspected is placed on a sample table 1, and power supply 4 for lamp causes two flash lamps 3 to radiate high power heat ray on the surface of C/C composite material 2. Radiation energy generating from the C/C complex, being synchronized with the heat ray radiation, is entered into an infrared thermo-graphic device and entered at higher rate into a central processing unit (CPU) 6 as thermal image data. CPU 6 regenerates the input image, shows time-sequence information on surface temperature distribution of the C/C composite material 2, determines its time dependence, and detects defects such as crack, separation, or hole in the complex 2.

LEGAL STATUS

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CLAIMS

[Claim(s)]
[Claim 1] the nondestructive inspection of the carbon fiber consolidation graphite composite which an exposure be synchronize with the heat radiation of a stop and a flash lamp , and carry out the image input of the thermal radiation energy from the carbon fiber consolidation graphite composite front face at the time of thermal unsteady with infrared thermograph equipment after irradiate the heat ray of a flash lamp on the front face of a carbon fiber consolidation graphite composite and heat in an instant , and be characterize by to detect a defective part by reproduce an input image and measure time amount change of skin temperature distribution .

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the nondestructive inspection of the C/C composite which can detect efficiently defective parts, such as a crack in the interior of a carbon fiber consolidation graphite composite (henceforth "C/C composite"), exfoliation, and a hole, with high degree of accuracy.
[0002]
[Description of the Prior Art] Composite molding of the carbon fiber is carried out with matrix resin, and since it has the thermal resistance and chemical stability which were excellent also in the pyrosphere which has high specific strength and a high specific Young's modulus, and exceeds 1000 degrees C, useful [of the C/C composite which hardened and carbonized / baking] is carried out as a structural material which begins the object for aerospace and is used by the elevated-temperature severe condition.

[0003] The fiber object which carried out orientation of the textile fabrics, such as plain weave manufactured from various raw materials, such as a polyacrylonitrile system, a rayon system, and a pitch system, satin, and twill, in the single dimension or the direction of many dimensions, the felt, a tow, etc. are used for the carbon fiber used as the reinforcement of C / C composite, and thermoplastics like the liquefied thermosetting resin of high carbonization nature, such as a phenol system and a furan system, and a tar pitch as matrix resin is used for it. The carbon fiber was fully soaked in matrix resin with means, such as impregnation and spreading, carries out after semi-hardening, creates prepreg, a laminating and after pressurizing and carrying out composite molding, it heats this prepreg in temperature of 100-250 degrees C, and it hardens a resinous principle thoroughly. C/C composite is manufactured by heating the acquired carbon fiber-resin compound hardening Plastic solid in temperature of 1000 degrees C or more in non-oxidizing atmospheres, such as nitrogen and an argon, and carrying out baking carbonization.
[0004] Thus, in each heat treatment process at the time of manufacture, gas components, such as a low-boiling point component in matrix resin and condensation water, remain, a hole is formed, the carbide of matrix resin exfoliates from a carbon fiber, or the C/C composite manufactured has the difficulty which defective parts, such as a crack, generate in the gap of the carbon fiber by which the laminating was carried out.
[0005] Generally, there are a coin tapping method by change of a tap tone and a supersonic method by change of the reflected sound of a supersonic wave as a nondestructive inspection which detects the construction material defective part of carbon material. However, a coin tapping method is based on an acoustic sense, since the element influenced by an experience and intuition is large, it is difficult to obtain objective inspection data, and there is a fault in which the time amount which inspection takes, and an effort increase. Furthermore, it is in the manufacture process of C/C composite, for example, in an initial burned product, by porosity, since reinforcement is low, according to the coin tapping method, a tap tone is absorbed, and it is hard to sense change of a sound, and there is also a possibility of destroying. On the other hand, since the area made into a subject of examination is small, measurement

efficiency becomes low, and in the initial burned product of C/C composite, since, and attenuation of a supersonic wave is very large, a supersonic method has a difficulty undetectable to accuracy. Furthermore, there is also a problem whose detection becomes impossible about the defect near the front face of C/C composite. Therefore, it is difficult to apply to the nondestructive inspection which detects the defective part of C/C composite by these inspection approaches.

[0006] Recently, the technique which measures the skin temperature of various matter and skin temperature distribution using infrared thermograph equipment is developed, and it is used widely. There is a method of detecting the difference in the description in the interior of the matter using change of the skin temperature distribution which appears based on a difference of the thermal property inside the matter in one of the applied technology of the, and it is utilized as a means to detect the abnormality section in construction material inside [various] an ingredient in un-destroying.

[0007] However, the thermometry by infrared thermograph equipment has the fault which produces a measurement error with the emissivity of a measured material-list side, the coating of high emissivity is applied to a measured material-list side, or processing of split-face-izing a measured material-list side is needed. In order to cancel such a fault, so, to JP,7-35620,A In the nondestructive inspection approach of measuring skin temperature distribution of a device under test using infrared thermograph equipment, and detecting the defect inside a device under test from this skin temperature distribution The high emissivity layer which consists of an ingredient with 0.2 or more emissivity between an infrared sensor and a device under test, The transparency layer which is a layer which consists of an infrared transparency ingredient is allotted so that said high emissivity layer may be located in a device-under-test side and said transparency layer may be located in an infrared sensor side. The band cut-off filter from which the wavelength band containing the specific absorption wavelength of the infrared radiation of said infrared transparency layer which is furthermore in the detection wavelength band of infrared thermograph equipment is removed is arranged between the infrared sensors of a transparency layer and infrared thermograph equipment. It lets these layers pass, skin temperature distribution of a device under test is measured, and the nondestructive inspection approach characterized by detecting a defect is proposed.

[0008] Moreover, these people did the pressure welding of the reticulum of high emissivity to the measured ingredient side, after they irradiated the heat ray with the flash lamp and heated from the upper part in an instant, developed the nondestructive inspection of the construction-material defective part characterized by to detect the construction material defective part of a measured ingredient for an exposure a stop and by subsequently measuring skin temperature distribution of a measured ingredient using infrared thermograph equipment, and proposed as Japanese Patent Application No. No. 296061 [seven to].

[0009]

[Problem(s) to be Solved by the Invention] this invention persons found out how the defective part which exists in the interior of C/C composite is promptly [exactly and] detectable, as a result of advancing research further for the C/C composite which has emissivity high in construction material based on the technique of above-mentioned Japanese Patent Application No. No. 296061 [seven to].

[0010] This invention is what was completed based on this knowledge, and that object is in offering the nondestructive inspections of the C/C composite which can detect defective parts, such as a crack inside C/C composite, exfoliation, and a hole, with an efficiently and sufficient precision also including intermediate products in the manufacture process of C/C composite, such as a carbon fiber-resin compound hardening Plastic solid and a medium burned product, including C/C composite.

[0011]

[Means for Solving the Problem]

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TECHNICAL FIELD

[Field of the Invention] This invention relates to the nondestructive inspection of the C/C composite which can detect efficiently defective parts, such as a crack in the interior of a carbon fiber consolidation graphite composite (henceforth "C/C composite"), exfoliation, and a hole, with high degree of accuracy.

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PRIOR ART

[Description of the Prior Art] Composite molding of the carbon fiber is carried out with matrix resin, and since it has the thermal resistance and chemical stability which were excellent also in the pyrosphere which has high specific strength and a high specific Young's modulus, and exceeds 1000 degrees C, useful [of the C/C composite which hardened and carbonized / baking] is carried out as a structural material which begins the object for aerospace and is used by the elevated-temperature severe condition.

[0003] The fiber object which carried out orientation of the textile fabrics, such as plain weave manufactured from various raw materials, such as a polyacrylonitrile system, a rayon system, and a pitch system, satin, and twill, in the single dimension or the direction of many dimensions, the felt, a tow, etc. are used for the carbon fiber used as the reinforcement of C / C composite, and thermoplastics like the liquefied thermosetting resin of high carbonization nature, such as a phenol system and a furan system, and a tar pitch as matrix resin is used for it. The carbon fiber was fully soaked in matrix resin with means, such as impregnation and spreading, carries out after semi-hardening, creates prepreg, a laminating and after pressurizing and carrying out composite molding, it heats this prepreg in temperature of 100-250 degrees C, and it hardens a resinous principle thoroughly. C/C composite is manufactured by heating the acquired carbon fiber-resin compound hardening Plastic solid in temperature of 1000 degrees C or more in non-oxidizing atmospheres, such as nitrogen and an argon, and carrying out baking carbonization.
[0004] Thus, in each heat treatment process at the time of manufacture, gas components, such as a low-boiling point component in matrix resin and condensation water, remain, a hole is formed, the carbide of matrix resin exfoliates from a carbon fiber, or the C/C composite manufactured has the difficulty which defective parts, such as a crack, generate in the gap of the carbon fiber by which the laminating was carried out.
[0005] Generally, there are a coin tapping method by change of a tap tone and a supersonic method by change of the reflected sound of a supersonic wave as a nondestructive inspection which detects the construction material defective part of carbon material. However, a coin tapping method is based on an acoustic sense, since the element influenced by an experience and intuition is large, it is difficult to obtain objective inspection data, and there is a fault in which the time amount which inspection takes, and an effort increase. Furthermore, it is in the manufacture process of C/C composite, for example, in an initial burned product, by porosity, since reinforcement is low, according to the coin tapping method, a tap tone is absorbed, and it is hard to sense change of a sound, and there is also a possibility of destroying. On the other hand, since the area made into a subject of examination is small, measurement efficiency becomes low, and in the initial burned product of C/C composite, since, and attenuation of a supersonic wave is very large, a supersonic method has a difficulty undetectable to accuracy. Furthermore, there is also a problem whose detection becomes impossible about the defect near the front face of C/C composite. Therefore, it is difficult to apply to the nondestructive inspection which detects the defective part of C/C composite by these inspection approaches.
[0006] Recently, the technique which measures the skin temperature of various matter and skin

temperature distribution using infrared thermograph equipment is developed, and it is used widely. There is a method of detecting the difference in the description in the interior of the matter using change of the skin temperature distribution which appears based on a difference of the thermal property inside the matter in one of the applied technology of the, and it is utilized as a means to detect the abnormality section in construction material inside [various] an ingredient in un-destroying.

[0007] However, the thermometry by infrared thermograph equipment has the fault which produces a measurement error with the emissivity of a measured material-list side, the coating of high emissivity is applied to a measured material-list side, or processing of split-face-izing a measured material-list side is needed. Then, in order to cancel such a fault, it is in JP,7-35620,A, In the nondestructive inspection approach of measuring skin temperature distribution of a device under test using infrared thermograph equipment, and detecting the defect inside a device under test from this skin temperature distribution The high emissivity layer which consists of an ingredient with 0.2 or more emissivity between an infrared sensor and a device under test, The transparency layer which is a layer which consists of an infrared transparency ingredient is allotted so that said high emissivity layer may be located in a device-under-test side and said transparency layer may be located in an infrared sensor side. The band cut-off filter from which the wavelength band containing the specific absorption wavelength of the infrared radiation of said infrared transparency layer which is furthermore in the detection wavelength band of infrared thermograph equipment is removed is arranged between the infrared sensors of a transparency layer and infrared thermograph equipment. It lets these layers pass, skin temperature distribution of a device under test is measured, and the nondestructive inspection approach characterized by detecting a defect is proposed.

[0008] Moreover, these people did the pressure welding of the reticulum of high emissivity to the measured ingredient side, after they irradiated the heat ray with the flash lamp and heated from the upper part in an instant, developed the nondestructive inspection of the construction-material defective part characterized by to detect the construction material defective part of a measured ingredient for an exposure a stop and by subsequently measuring skin temperature distribution of a measured ingredient using infrared thermograph equipment, and proposed as Japanese Patent Application No. No. 296061 [seven to].

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EFFECT OF THE INVENTION

[Effect of the Invention] The heat ray which was irradiated by the C/C composite front face according to the nondestructive inspection of the C/C composite of this invention the above passage It is possible to detect efficiently defective parts, such as a crack in the interior of C/C composite, exfoliation, and a hole, with high degree of accuracy by carry out the image input of the thermal radiation energy from C/C composite with infrared thermograph equipment at the time of thermal unsteady [in the radiation process of the heat energy from the C/C composite front face heated in an instant], reproduce an input image, and measure time amount change of skin temperature distribution. Furthermore, a defective part can be detected by un-destroying also about the case where an oxidation-resistant coat and an oxidation-resistant carbonaceous coat are formed in the intermediate product and C/C composite front face in the manufacture process of C/C composite, and it is very useful as detection method of production control.

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] this invention persons found out how the defective part which exists in the interior of C/C composite is promptly [exactly and] detectable, as a result of advancing research further for the C/C composite which has emissivity high in construction material based on the technique of above-mentioned Japanese Patent Application No. No. 296061 [seven to]. [0010] This invention is what was completed based on this knowledge, and that object is in offering the nondestructive inspections of the C/C composite which can detect defective parts, such as a crack inside C/C composite, exfoliation, and a hole, with an efficiently and sufficient precision also including intermediate products in the manufacture process of C/C composite, such as a carbon fiber-resin compound hardening Plastic solid and a medium burned product, including C/C composite.

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MEANS

[Means for Solving the Problem] The nondestructive inspection of the carbon fiber consolidation graphite composite by this invention for attaining the above-mentioned object After irradiating the heat ray of a flash lamp on the front face of C/C composite and heating in an instant, an exposure A stop, Make it synchronize with the heat radiation of a flash lamp, and the image input of the thermal radiation energy from the C/C composite front face at the time of thermal unsteady is carried out with infrared thermograph equipment. It is characterized by detecting a defective part on a configuration by reproducing an input image and measuring time amount change of skin temperature distribution.
[0012]
[Embodiment of the Invention] It is possible to apply to the compound hardening Plastic solid of the carbon fiber and matrix resin which are not only in C/C composite but in a manufacture process at the target C/C composite, or the baking carbonization article of a midcourse phase with the nondestructive inspection of this invention.
[0013] After giving a thermal load to C/C composite, if the skin temperature distribution is measured, it will originate in defective parts, such as a crack which exists in the interior of C/C composite, exfoliation, and a hole, and change will arise in the temperature distribution of C / C composite front face. For example, if an exposure is stopped after irradiating a pulse-like heat ray momentarily with a flash lamp on the front face of C/C composite and heating a front face uniformly in an instant In the time of thermal unsteady until the heat which entered from the front face will transmit the interior of C/C composite, will arrive at a rear face and will be in equilibrium thermally soon A heat transfer rate changes with the defective parts which exist in the interior, and since the skin temperature of the field corresponding to a defective part and a normal field differs, a difference is produced to thermal radiation energy.
[0014] By using change of this skin temperature distribution at the time of thermal unsteady as a thermal image, it is made to synchronize with the heat radiation of a flash lamp, and an image input is carried out with infrared thermograph equipment at a processing unit (CPU) at high speed. If the inputted thermal image is reproduced with a processing unit (CPU), it outputs to an image display device and time amount change of temperature distribution is observed, corresponding to a location, magnitude, a configuration of a defective part, etc., it can continue broadly and a defective part can be detected visually. For example, since the opening formed in the defective part in the cooling process will delay heat transfer from a front face to the interior if defective parts, such as a crack, exfoliation, and a hole, are near [front face] this when heat energy is irradiated to a C/C composite front face in an instant, on a C/C composite front face, a high temperature field is relatively formed corresponding to a defective part. Therefore, when the temperature change and temperature distribution in a cooling process of a C/C composite front face are measured to time series using an infrared CCD detector, the time zone which becomes possible [catching a high temperature energy radiation zone relatively corresponding to a defective part] will exist. Thus, if the electrical signal of an infrared CCD detector is processed using an image processing system, a defective part can be detected as an image and inspection of a 200x200mm measurement field will be attained from the heat radiation of a flash lamp to detection of a defective part

by the time amount for dozens of seconds - about 1 minute.
[0015] Moreover, since emissivity is high, each intermediate product including C/C composite, such as a compound hardening Plastic solid of the carbon fiber and matrix resin in the manufacture process of C/C composite or a baking carbonization article of a midcourse phase, can measure skin temperature distribution with a sufficient precision. Therefore, it becomes possible about defective parts, such as a crack in the interior of C/C composite, exfoliation, and a hole, high degree of accuracy and to detect efficiently.

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EXAMPLE

[Example] Hereafter, the example of this invention is concretely explained as contrasted with the example of a comparison.
[0017] Examples 1-5, the example 1 of a comparison - 3 drawing 1 are the whole block diagrams which illustrated the equipment used for the nondestructive inspection of this invention. In the equipment of drawing 1 , on the sample base 1, the C/C composite 2 which is a charge of inspected material is laid, two flash lamps 3 of high power are arranged in the slanting upper part of the sample base 1, and a heat ray is irradiated by the front face of C / C composite 2 by high power according to the power source 4 for lamps. It is made to synchronize with the heat radiation of a flash lamp 3, the radiant energy from the C/C composite 2 is inputted into the infrared thermograph equipment 5 arranged right above the sample base 1, and it inputs into a processing unit 6 (CPU) as heat image data at high speed. The inputted thermal image is reproduced with a processing unit 6 (CPU), a thermal image is displayed on an image display device 7, and image display of the time series information about skin temperature distribution of the C/C composite 2 is carried out.
[0018] The typical experiment was conducted by the following approach using above equipment. Production of C / C composite (inspection sample): It applied so that the volume fraction of a carbon fiber might become 60% at [by plain weave carbon fiber textile-fabrics [Toho Rayon Co., Ltd.] W6101] of a polyacrylonitrile system by using the initial condensate [PR940 made from Sumitomo DEYUREZU] of phenol resin as matrix resin, and it was air-dry for 48 hours, and the prepreg sheet was created. The laminating of these 16 prepreg sheets was carried out, and it put into the mould, and putting the pressure of 20 kg/cm2, at the temperature of 130 degrees C, subsequently, with the temperature of 250 degrees C, heating pressure treatment was carried out for 3 hours, and it hardened for 10 hours. This carbon fiber-resin compound hardening Plastic solid was put into the firing furnace held on a nitrogen-gas-atmosphere mind, with the programming rate of 20 degrees C/hr, it heated at 1000 degrees C and baking carbonization was carried out. Furthermore, it sank in, the initial condensate of furfuryl alcohol was again moved to the firing furnace, it heated to 2000 degrees C with the programming rate of 50 degrees C/hr, and 250mm of every direction, and C / C composite with a thickness of 4mm were produced. Thus, about the carbon fiber-resin compound hardening Plastic solid (sample A) in the production process of the produced C/C composite, 1000-degree-C burned product (sample B), and 2000-degree-C burned product (sample C), the opening section was beforehand formed in the position as a false defect, and it considered as the object sample of nondestructive inspection.
[0019] This sample A-C was carried on the sample base 1 of drawing 1 , the heat ray was irradiated with the flash lamp 3 for a moment, and it heated in temperature of 60-70 degrees C in an instant. Then, the image input of the radiant energy from sample A-C was carried out with infrared thermograph equipment 5 at the processing unit 6 (CPU), it reproduced, image display of the skin temperature distribution was carried out to the image display device 7, and the time amount change was measured.
[0020] Thus, the detection result of the obtained false defective part was judged in accordance with the following criterion, and it was shown in a table 1 with the time amount which inspection took the result. Moreover, the false defective part was detected with the supersonic method about sample A-C same for

a comparison, and the result was also carried jointly in a table 1.
O -- Detection good x -- Detection failure [0021]
[A table 1]

例No.	検査試料	疑似欠陥		検査結果	検査時間
		位置	空隙部		
実施例1	A	表面下0.5mm	φ10mm×0.2mm	○	30分／m²
実施例2	A	表面下 2mm	φ10mm×0.2mm	○	30分／m²
実施例3	B	表面下 1mm	φ10mm×0.2mm	○	30分／m²
実施例4	B	表面下 2mm	φ10mm×0.2mm	○	30分／m²
実施例5	C	表面下1.5mm	φ10mm×0.2mm	○	30分／m²
比較例1	A	表面下 2mm	φ10mm×0.2mm	○	5時間／m²
比較例2	B	表面下 2mm	φ10mm×0.2mm	×	5時間／m²
比較例3	C	表面下1.5mm	φ10mm×0.2mm	○	5時間／m²

[0022] Often [it is short and] inspecting is possible, and the time amount which can detect clearly the defective part which each formed in false in the example, and inspection takes from the result of a table 1 can also be further detected also about the carbon fiber-resin compound hardening Plastic solid (sample A) and medium burned product (sample B) not only in C/C composite (sample C) but the manufacture process. On the other hand, the time amount which inspection takes in the example of a comparison was long, and was not able to detect a defective part clearly by Sample B.

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] It is the whole block diagram which illustrated the equipment used for the nondestructive inspection which detects the defective part of the C/C composite concerning this invention.
[Description of Notations]
1 Sample Base
2 C/C Composite
3 Flash Lamp
4 Power Source for Lamps
5 Infrared Thermograph Equipment
6 Processing Unit (CPU)
7 Image Display Device

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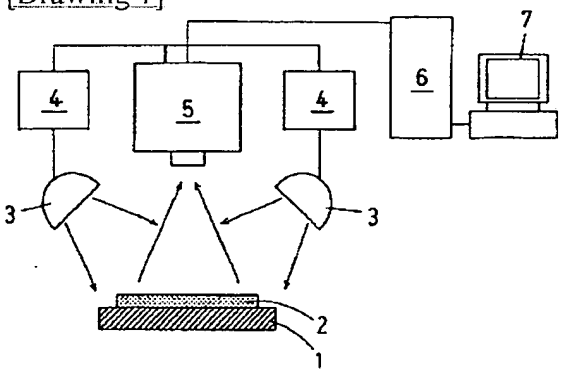
DRAWINGS

[Drawing 1]



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特開平10－96705

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// Ｃ０４Ｂ　35/83		Ｃ０４Ｂ　35/52		Ｅ

審査請求　未請求　請求項の数１　ＦＤ　（全５頁）

(21)出願番号　特願平8－273038

(22)出願日　平成８年(1996) ９月24日

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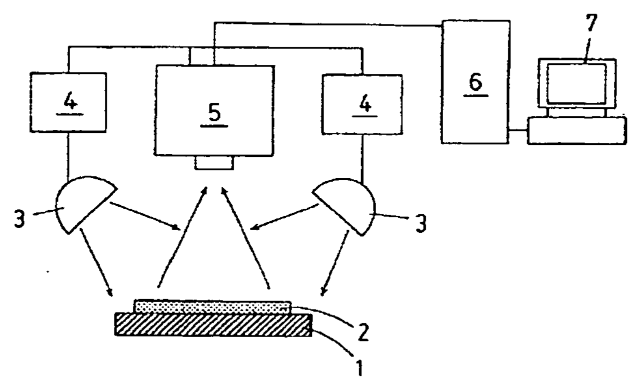
(74)代理人　弁理士　福田　保夫　（外１名）

(54)【発明の名称】　炭素繊維強化炭素複合材の非破壊検査法

(57)【要約】
【課題】　熱的非定常時におけるＣ／Ｃ複合材表面の温度分布の時間変化を測定することにより、Ｃ／Ｃ複合材内部の亀裂、剥離、空孔などの欠陥部位を高精度で能率的に検出することのできるＣ／Ｃ複合材の非破壊検査法を提供する。
【解決手段】　Ｃ／Ｃ複合材にフラッシュランプの熱線を照射して瞬時に加熱したのち照射を止め、フラッシュランプの熱線照射に同期させて熱的非定常時におけるＣ／Ｃ複合材表面からの熱放射エネルギーを赤外線サーモグラフ装置により画像入力し、入力画像を再生して表面温度分布の時間変化を測定することによりＣ／Ｃ複合材内部の欠陥部位を検出する。



【特許請求の範囲】
【請求項１】　炭素繊維強化炭素複合材の表面にフラッシュランプの熱線を照射して瞬時に加熱したのち照射を止め、フラッシュランプの熱線照射に同期させて熱的非定常時における炭素繊維強化炭素複合材表面からの熱放射エネルギーを赤外線サーモグラフ装置により画像入力し、入力画像を再生して表面温度分布の時間変化を測定することにより欠陥部位を検出することを特徴とする炭素繊維強化炭素複合材の非破壊検査法。
【発明の詳細な説明】
【０００１】
【発明の属する技術分野】本発明は、炭素繊維強化炭素複合材（以下、「Ｃ／Ｃ複合材」という。）の内部における亀裂、剥離、空孔などの欠陥部位を高精度で能率的に検出することのできるＣ／Ｃ複合材の非破壊検査法に関する。
【０００２】
【従来の技術】炭素繊維をマトリックス樹脂とともに複合成形し、硬化および焼成炭化したＣ／Ｃ複合材は、高い比強度や比弾性率を有し、また１０００℃を越える高温域においても優れた耐熱性および化学的安定性を備えているため、航空宇宙用をはじめ高温過酷な条件で使用される構造材料として有用されている。
【０００３】Ｃ／Ｃ複合材の強化材となる炭素繊維には、ポリアクリロニトリル系、レーヨン系、ピッチ系などの各種原料から製造された平織、朱子織、綾織などの織布を一次元または多次元方向に配向した繊維体、フェルト、トウなどが使用され、マトリックス樹脂としてはフェノール系、フラン系など高炭化性の液状熱硬化性樹脂、タールピッチのような熱可塑性物質が用いられる。炭素繊維は、含浸、塗布などの手段によりマトリックス樹脂で十分に濡らしたのち半硬化してプリプレグを作成し、このプリプレグを積層、加圧して複合成形したのち、１００～２５０℃の温度に加熱して樹脂成分を完全に硬化する。得られた炭素繊維－樹脂複合硬化成形体を、窒素、アルゴンなどの非酸化性雰囲気中で１０００℃以上の温度に加熱して焼成炭化することによりＣ／Ｃ複合材が製造される。
【０００４】このようにして製造されるＣ／Ｃ複合材は、製造時の各熱処理過程において、例えばマトリックス樹脂中の低沸点成分や縮合水などのガス状成分が残留して空孔が形成されたり、マトリックス樹脂の炭化物が炭素繊維から剥離したり、積層された炭素繊維の間隙に亀裂などの欠陥部位が発生する難点がある。
【０００５】一般に、炭素材の材質欠陥部位を検出する非破壊検査法として、打音の変化によるコインタッピング法や超音波の反射音の変化による超音波法がある。しかしながら、コインタッピング法は聴覚によるものであり、経験や勘に左右される要素が大きいため客観的な検査データを得ることが難しく、また検査に要する時間や労力が増大する欠点がある。更に、Ｃ／Ｃ複合材の製造過程にある、例えば初期焼成品では多孔質で強度が低いためにコインタッピング法によると打音が吸収されて音の変化を感知しにくく、また破壊するおそれもある。一方、超音波法は検査対象とする面積が小さいので測定能率が低くなり、またＣ／Ｃ複合材の初期焼成品では多孔質なため超音波の減衰が非常に大きいため正確に検出することができない難点がある。更に、Ｃ／Ｃ複合材の表面近傍にある欠陥については検出が不可能となる問題もある。したがって、これらの検査方法ではＣ／Ｃ複合材の欠陥部位を検出する非破壊検査に適用することは困難である。
【０００６】近時、各種物質の表面温度や表面温度分布を赤外線サーモグラフ装置を用いて測定する技術が開発され、広く利用されている。その応用技術の一つに、物質内部の熱的特性の相違に基づいて現れる表面温度分布の変化を利用して物質内部における性状の差異を検出する方法があり、各種材料内部の材質異常部を非破壊的に検出する手段として活用されつつある。
【０００７】しかしながら、赤外線サーモグラフ装置による温度測定は被測定材料表面の放射率によって測定誤差を生じる欠点があり、高放射率の塗料を被測定材料表面に塗布したり被測定材料表面を粗面化するなどの処理が必要とされている。そこで、このような欠点を解消するために、例えば特開平７－３５６２０号公報には、赤外線サーモグラフ装置を用いて被測定物の表面温度分布を測定し、該表面温度分布から被測定物内部の欠陥を検出する非破壊検査方法において、赤外線センサーと被測定物との間に、０．２以上の放射率をもつ材料から成る高放射率層と、赤外線透過材料から成る層である透過層とを、前記高放射率層が被測定物側に位置し前記透過層が赤外線センサー側に位置するように配して、さらに赤外線サーモグラフ装置の検出波長帯域内にある前記赤外線透過層の赤外線の特定吸収波長を含む波長帯域を除去するバンドカットフィルターを透過層と赤外線サーモグラフ装置の赤外線センサー間に配設し、これらの層を通して、被測定物の表面温度分布を測定し、欠陥を検出することを特徴とする非破壊検査方法が提案されている。
【０００８】また、本出願人は、高放射率の網状体を被測定材料面に圧接して、その上方からフラッシュランプにより熱線を照射し瞬時に加熱したのち照射を止め、次いで赤外線サーモグラフ装置を用いて被測定材料の表面温度分布を測定することにより被測定材料の材質欠陥部位を検知することを特徴とする材質欠陥部の非破壊検査法を開発し、特願平７－２９６０６１号として提案した。
【０００９】
【発明が解決しようとする課題】本発明者らは、上記特願平７－２９６０６１号の技術を基に、材質的に高い放射率を有するＣ／Ｃ複合材を対象にして更に研究を進め

た結果、Ｃ／Ｃ複合材内部に存在する欠陥部位を的確かつ迅速に検出できる方法を見いだした。

【００１０】本発明はこの知見に基づいて完成したもので、その目的はＣ／Ｃ複合材をはじめ、Ｃ／Ｃ複合材の製造過程にある炭素繊維－樹脂複合硬化成形体や中間焼成品などの中間製品も含めて、Ｃ／Ｃ複合材内部の亀裂、剥離、空孔などの欠陥部位を能率的に、かつ精度よく検出することのできるＣ／Ｃ複合材の非破壊検査法を提供することにある。

【００１１】

【課題を解決するための手段】上記の目的を達成するための本発明による炭素繊維強化炭素複合材の非破壊検査法は、Ｃ／Ｃ複合材の表面にフラッシュランプの熱線を照射して瞬時に加熱したのち照射を止め、フラッシュランプの熱線照射に同期させて熱的非定常時におけるＣ／Ｃ複合材表面からの熱放射エネルギーを赤外線サーモグラフ装置により画像入力し、入力画像を再生して表面温度分布の時間変化を測定することにより欠陥部位を検出することを構成上の特徴とする。

【００１２】

【発明の実施の形態】本発明の非破壊検査法で対象とするＣ／Ｃ複合材には、Ｃ／Ｃ複合材ばかりではなく製造過程にある炭素繊維とマトリックス樹脂との複合硬化成形体、あるいは中間段階の焼成炭化品などにも適用することが可能である。

【００１３】Ｃ／Ｃ複合材に熱的負荷を与えたのち、その表面温度分布を測定するとＣ／Ｃ複合材の内部に存在する亀裂、剥離、空孔などの欠陥部に起因してＣ／Ｃ複合材表面の温度分布に変化が生じる。例えば、Ｃ／Ｃ複合材の表面にフラッシュランプにより瞬間的にパルス状の熱線を照射して瞬時に表面を一様に加熱したのち照射を止めると、表面から入った熱がＣ／Ｃ複合材内部を伝達して裏面に到達し、やがて熱的に平衡状態になるまでの熱的非定常時において、内部に存在する欠陥部位により熱伝達速度が変化して、欠陥部位に対応した領域と正常な領域との表面温度が異なるために熱放射エネルギーに差異を生じる。

【００１４】この熱的非定常時における表面温度分布の変化を熱画像として、フラッシュランプの熱線照射に同期させて赤外線サーモグラフ装置により演算処理装置（ＣＰＵ）に高速で画像入力する。入力された熱画像を演算処理装置（ＣＰＵ）で再生して画像表示装置に出力し、温度分布の時間変化を観察すれば欠陥部位の位置や大きさおよび形状などに対応して、広範囲に亘って欠陥部位を目視にて検出することができる。例えば、Ｃ／Ｃ複合材表面に対して瞬時に熱エネルギーを照射した場合、該表面近傍に亀裂、剥離、空孔などの欠陥部位があると、冷却過程において欠陥部位に形成された空隙が表面から内部への熱伝達を遅延させるので、Ｃ／Ｃ複合材表面では欠陥部位に対応して相対的に高温度領域が形成される。したがって、赤外ＣＣＤ検知器を用いてＣ／Ｃ複合材表面の冷却過程での温度変化および温度分布を時系列に測定すると、欠陥部位に対応して相対的に高熱エネルギー放射領域を捉えることが可能となる時間帯が存在することとなる。このようにして、赤外ＣＣＤ検知器の電気信号を画像処理装置を用いて処理を行うと欠陥部位を画像として検出することができ、フラッシュランプの熱線照射から欠陥部位の検出まで数十秒～１分程度の時間で、２００×２００㎜の測定領域の検査が可能となる。

【００１５】また、Ｃ／Ｃ複合材をはじめ、Ｃ／Ｃ複合材の製造過程にある炭素繊維とマトリックス樹脂との複合硬化成形体あるいは中間段階の焼成炭化品などの中間製品は、いずれも放射率が高いので精度よく表面温度分布を測定することができる。したがって、Ｃ／Ｃ複合材内部における亀裂、剥離、空孔などの欠陥部位を高精度かつ能率的に検出することが可能となる。

【００１６】

【実施例】以下、本発明の実施例を比較例と対比して具体的に説明する。

【００１７】実施例１～５、比較例１～３

図１は本発明の非破壊検査法に用いた装置を例示した全体構成図である。図１の装置において、試料台１の上には被検査材料であるＣ／Ｃ複合材２が載置され、試料台１の斜め上方には高出力のフラッシュランプ３が２個配置されており、ランプ用電源４により高出力で熱線がＣ／Ｃ複合材２の表面に照射される。フラッシュランプ３の熱線照射に同期させてＣ／Ｃ複合材２からの放射エネルギーが、試料台１の真上に配置された赤外線サーモグラフ装置５に入力され、演算処理装置６（ＣＰＵ）に高速で熱画像データとして入力する。入力された熱画像を演算処理装置６（ＣＰＵ）で再生し、画像表示装置７に熱画像が表示され、Ｃ／Ｃ複合材２の表面温度分布に関する時系列情報が画像表示される。

【００１８】上記の装置を用いて、下記の方法により模式的な実験を行った。

Ｃ／Ｃ複合材（検査試料）の作製：ポリアクリロニトリル系の平織炭素繊維織布〔東邦レーヨン（株）製 W6101〕にフェノール樹脂初期縮合物〔住友デュレズ（株）製 PR940〕をマトリックス樹脂として炭素繊維の体積含有率が６０％となるように塗布し、４８時間風乾してプリプレグシートを作成した。このプリプレグシート１６枚を積層してモールドに入れ、２０Kg/cm²の圧力を掛けながら温度１３０℃で１０時間、次いで温度２５０℃で３時間加熱加圧処理して硬化した。この炭素繊維－樹脂複合硬化成形体を窒素ガス雰囲気に保持した焼成炉に入れ、２０℃／hrの昇温速度で１０００℃に加熱して焼成炭化した。更に、フルフリルアルコール初期縮合物を含浸し、再び焼成炉に移して５０℃／hrの昇温速度で２０００℃まで加熱して、縦横２５０㎜、厚さ４㎜のＣ／Ｃ

複合材を作製した。このようにして作製したＣ／Ｃ複合材の作製過程にある、炭素繊維－樹脂複合硬化成形体（試料Ａ）、１０００℃焼成品（試料Ｂ）、２０００℃焼成品（試料Ｃ）について、予め所定の位置に疑似欠陥として空隙部を形成し、非破壊検査の対象試料とした。

【００１９】この試料Ａ～Ｃを図１の試料台１の上に載せ、フラッシュランプ３により一瞬熱線を照射して瞬時に６０～７０℃の温度に加熱した。その後、試料Ａ～Ｃからの放射エネルギーを赤外線サーモグラフ装置５により演算処理装置６（ＣＰＵ）に画像入力し、再生して画像表示装置７にその表面温度分布を画像表示して、その時間変化を測定した。

【００２０】このようにして得られた疑似欠陥部の検出結果を、下記の判定基準にしたがって判定し、その結果を検査に要した時間とともに表１に示した。また、比較のために同一の試料Ａ～Ｃについて超音波法で疑似欠陥部の検出を行い、その結果も表１に併載した。

○…検出可

×…検出不可

【００２１】

【表１】

例No.	検査試料	疑似欠陥		検査結果	検査時間
		位置	空隙部		
実施例１	Ａ	表面下0.5㎜	φ10㎜×0.2㎜	○	30分／㎡
実施例２	Ａ	表面下 2㎜	φ10㎜×0.2㎜	○	30分／㎡
実施例３	Ｂ	表面下 1㎜	φ10㎜×0.2㎜	○	30分／㎡
実施例４	Ｂ	表面下 2㎜	φ10㎜×0.2㎜	○	30分／㎡
実施例５	Ｃ	表面下1.5㎜	φ10㎜×0.2㎜	○	30分／㎡
比較例１	Ａ	表面下 2㎜	φ10㎜×0.2㎜	○	５時間／㎡
比較例２	Ｂ	表面下 2㎜	φ10㎜×0.2㎜	×	５時間／㎡
比較例３	Ｃ	表面下1.5㎜	φ10㎜×0.2㎜	○	５時間／㎡

【００２２】表１の結果から、実施例ではいずれも疑似的に形成した欠陥部位を明確に検出することができ、また検査に要する時間も短く能率よく検査することが可能であり、更に、Ｃ／Ｃ複合材（試料Ｃ）のみでなく、その製造過程にある炭素繊維－樹脂複合硬化成形体（試料Ａ）や中間焼成品（試料Ｂ）についても検出することができる。これに対して、比較例では検査に要する時間が長く、また試料Ｂでは明確に欠陥部位を検出することができなかった。

【００２３】

【発明の効果】以上のとおり、本発明のＣ／Ｃ複合材の非破壊検査法によれば、Ｃ／Ｃ複合材表面に照射された熱線により瞬時に加熱されたＣ／Ｃ複合材表面からの熱エネルギーの放射過程にある熱的非定常時において、Ｃ／Ｃ複合材からの熱放射エネルギーを赤外線サーモグラフ装置により画像入力し、入力画像を再生して表面温度分布の時間変化を測定することによりＣ／Ｃ複合材内部にある亀裂、剥離、空孔などの欠陥部位を高精度で能率的に検出することが可能である。更に、Ｃ／Ｃ複合材の製造過程にある中間製品やＣ／Ｃ複合材表面に耐酸化性の被膜や炭素質被膜を形成した場合についても非破壊で欠陥部位を検出可能であり、工程管理の検査法として極めて有用である。

【図面の簡単な説明】

【図１】本発明に係るＣ／Ｃ複合材の欠陥部位を検出する非破壊検査法に用いられる装置を例示した全体構成図である。

【符号の説明】

１　試料台

２　Ｃ／Ｃ複合材

３　フラッシュランプ

４　ランプ用電源

５　赤外線サーモグラフ装置

６　演算処理装置（ＣＰＵ）

７　画像表示装置

【図１】